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GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT INITIATION

Date: 1/9/79

Project Title: Small Angle Neutron Scattering Studies of Materials.

Green card

Project No: E-19-676 (continuation of E-19-660)

Project Director: Dr. S. Spooner

Sponsor: Union Carbide Corporation, Nuclear Division

Agreement Period: From 12/1/78 Until 11/30/79

Type Agreement: Subcontract No. 7449 (under DOE Prime W-7405-eng-26)

Amount: $30,493

Reports Required: Informal Progress Reports, Final Report

Sponsor Contact Person (s):

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(thru OCA)

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Defense Priority Rating: N/A

Assigned to: Chemical Engineering (School/Laboratory)

COPIES TO:

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- Other

CA-3 (3/76)

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION

Date: June 11, 1980

Project Title: Small Angle Neutron Scattering Studies of Materials.

Project No: E-19-676 NOTE: Continuation of E-19-660

Project Director: Dr. S. Spooner

Sponsor: Union Carbide Corporation, Nuclear Division

Effective Termination Date: 11/30/79

Clearance of Accounting Charges: 11/30/79

Grant/Contract Closeout Actions Remaining:

x Final Invoice and Closing Documents
_ Final Fiscal Report
x Final Report of Inventions
x Govt. Property Inventory & Related Certificate
_ Classified Material Certificate
_ Other ____________

Assigned to: Chemical Engineering (School/~~Laboratory~~)

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CA-4 (1/79)

E-19-616

SMALL ANGLE NEUTRON SCATTERING OF MATERIALS

Progress Report on Subcontract
No. 7449

Report Period January 1, 1978
to October 31, 1978

telephone
x 3013

Stepehen Spooner (031-28-2085)

Georgia Institute of Technology

Review of Project Activity

Small angle neutron scattering (SANS) work consisted of specific scattering experimentation, development of instrumentation, preparation of research publications and a presentation at the Denver, Colorodo AIME Meeting. Recommendations were made on the development of the ORNL facility and groundwork was laid for new experiments with irradiation damage research.

Scattering Research

The contribution to the Solid State Division report describes current findings on the deformation-induced magnetic martensite in 304 stainless steel. Transmission electron microscopy was done on selected samples of the sample material for the purpose of defining probable morphology of the magnetic phase. Further work on the magnetic scattering from these stainless steel samples is needed to make a definitive analysis of structure.

Spinodally decomposition in Cu 4% Ti was the subject of a SANS study in which aging time-dependence on composition modulation wavelength. While it is generally expected the decomposition progresses extensively during continuous cooling, the dominance of the structure was not expected. A second method of sample preparation proved ineffective in suppressing the extensive spinodal decomposition. Until this problem can be solved, further scattering work is not regarded as productive.

A survey of SANS effects in several commercial alloys showed that no large scattering intensity will be found. Transmission electron microscopy done on samples of the alloys showed large precipitates (∿5000Å) whose scattering effects can only be seen at very small scattering angles.

A preliminary investigation of SANS scattering from an Invor-type FeNi alloy was undertaken. A permanent magnet exerting a field of 4700 Gauss at the gap center was sufficient to exert a surprising control of very small angle scattering. In particular, there was a significant supression of scattering caused by the magnetic field. However, the apparent direction of asymmetry was "wrong" in the sense that scattering along the field axis was not enhanced. Similar effects were seen in Armco iron. These effects can be seen in the deformed stainless steel. A possible source of these interesting effects may be a magnetic domain wall structure response to field.

Finally, there continues to be a problem in the quantitative standardization of scattering intensity. With the object of relieving our dependence on a vanadium scattering standard which gives too weak a scattering for our experiment, a standard glassy carbon sample and a previously calibrated aluminum void scattering were measured and inter-comparison was satisfactory (within 20%). However, the principal drawback in these calibrations is that no measure of counter uniformity can conveniently be assessed without tedious point-by-point measurements.

Instrumental Considerations

A compact lead gamma-ray shield was assembled for the purpose of reactor-irradiation void sample SANS experiments. A simple method of handling micro-tensile specimen pieces was devised. It is intended to use this system in studies of niobium-zirconium alloy void structure.

Several instrumentation recommendations were made for improvement of signal-to-noise in the ORNL SANS machine. Utilization of the MODCOMP system for any data processor improvement was strongly recommended.

Papers in Preparation

Three short papers presented at the International Small Angle Scattering Meeting are being rewritten for publication following reviewers' criticisms of the original manuscripts. Principal parts being worked on are (1) extension of the ORNL SANS instrument description to emphasize unique design features, (2) re-orienting the direction of analysis of niobium void structure and (3) incorporation of new SANS data on irradiated stainless steel.

Presentations

A poster presentation on the application of SANS in metal and alloy studies was made at the Denver AIME Meeting in March, 1978. This was done in collaboration with an x-ray SANS poster presentation.

Projects Under Consideration

Niobium void structure studies are being planned in cooperation with W.B. Wiffen of the Metallurgy and Ceramics Division.

SANS studies utilizing more carefully controlled magnetic fields are being planned where a neutron diffractometer electro magnet will be borrowed from Georgia Tech. Irradiated stainless steel, deformed stainless steel and several magnetic alloys are under consideration for these studies.

Respectfully submitted,

Stephen Spooner
Associate Professor
for Metallurgy

SS/es

E-19-616

SMALL ANGLE NEUTRON SCATTERING OF MATERIALS

Progress Report on Subcontract
No. 7449

Report Period December 1, 1978
to November 31, 1979

final?

Stephen Spooner (031-28-2085)

Georgia Institute of Technology

Summary

Small angle neutron scattering (SANS) activity on the SANS machine at ORR was directed more toward research and less toward machine development during the reporting period. Limits to machine performance were more quantitatively defined and special data analysis considerations appropriate to high cross section scattering problems were encountered. Four research efforts were undertaken which are expected to lead to publishable results: (1) analysis of void structure in niobium alloys, (2) search for magnetic SANS in irradiated 304 stainless steel, (3) analysis of the precipitation processes in Mn-40% Cu and (4) investigation of magnetic field effects on deformed stainless steel. Results of efforts (1) and (2) are being submitted for publication in a symposium on the Advanced Techniques for the Analysis of Microstructure.

Facility Development

During the reporting period the SANS facility at ORR was improved primarily in the area software and data handling capability. An electromagnet was installed and, with a shortening of the incident beam tube, the capability was added for investigating problems magnetic field at low temperature. No improvements were made in the incident beam intensity or background shielding.

The amount of research has increased on the ORR SANS machine as we have gained confidence in the machine's capabilities. The absolute calibration of scattering by measurement of the incident beam seems most applicable since the beam intensity is so low. Quantitative results have been obtained for high scattering cross section problems. However, multiple small angle scattering is very likely to occur and such possibilities will require evaluation.

While working on the analysis of scattering from voids in niobium and later when analysing magnetic field effects, we looked in detail at the 2-dimensional scattering data. Several experimental and instrumental problems came to our attention; (1) beam stop placement, (2) beam center determination, (3) position encoding proportionality, (4) electronic shifting and (5) electronic distortions. The points of the intensity distribution (3) through (5) may be found in the new large detector installed at HFIR since they are of electronic origin. The intensity distribution of a strong cylindrically symmetric scatterer such as carbon provides a convenient test data set in which to examine the distortions and shifts of the data due to the above mentioned effects. The simplest test consists in the examination of an azimuthal scan at fixed scattering angle. This scan will be independent of azimuthal angle only when the precise beam center is known, the beam stop is properly centered and the detector encoding proportionality is correctly. In general, azimuthal intensity variations will be sinesoidal - the minima and maxima will indicate the appropriate corrections in the center and proportionality factor for the x0 or y- direction on the detector. In one unfortunate case, there appeared to be a coupling between the x and y encoding proportionality constant. This appeared to shear the cylindrically symmetric intensity distribution.

Research Progress

A SANS structure analysis of the void structure of reactor irradiated niobium and niobium alloys was undertaken. SANS is a non-destructive method and x-ray small angle scattering proved to be a virtually impossible experiment due to radioactivity background. Although SANS required background correction for gamma radiation, neutron scattering can be an ambiguously differentiated from gamma ray scattering through use of neutron absorbing filters. After much detailed data examination, only qualitative trends in the void structure could be found. Clearly the ORR SANS machine as presently arranged is not adequate for a quantitative void analysis in this problem. The principal limitation was the small scattering signal was only a little larger than the radioactivity background. Incident beam intensity improvements are needed in this case.

Work on irradiated stainless steel continued with an examination of magnetic field dependence in "void" scattering. No field dependence was found in the examined scattering angle range which indicates that magnetic material if present in stainless steel occured in precipitates of dimensions quite different from the void sizes. The measured scattering was calibrated with the incident beam intensity and void fraction was calculated from Qo, the integrated intensity. Multiple scattering effects were pointed out at the Las Vegas seminar as an explanation for the excessively large void fraction. This was not anticipated but should have been appreciated since our work can only be done in large scattering cross section problems where multiple scattering is very likely to occur.

SANS from Mn-40% Cu and Fe-Cr-Co-Si alloys resulted in interesting alloy structure analyses. In the manganese alloy which was heat treated over a range of temperatures, apparently two precipitation processes can occur. This alloy was suppled by E. R. Vance (now at Penn State) for preliminary analysis and we seem to have bracketed the times and temperatures within which further study of precipitation processes can be done. The Fe-Cr-Co-Si alloy proved to be an interesting magnetic alloy problem in which both nuclear and magnetic scattering density fluctuation give strong scattering at small angles. The interaction between the alloy composition and magnetic processes (domain wall pinning) could be readily explored with SANS.

Work on deformed stainless steel has continued with the exploration of magnetic field effects. The deformed structure is complex and very fine scaled so that the magnetic scattering interpretation may not be straight forward. Interesting results include the indication of a remanent magnetization and isotropy and either a strong refraction or a domain boundary scattering effect. Although this problem lacks elegance, results point to other (and probably better) experiments that can be done in technically interesting magnetic materials (perament magnets).

Recommendations for Instrument Development

Neutron intensity and instrument sheilding must be improved to make the ORR SANS useful to the Division. Emphasis on magnetic scattering capabilities could lead to a unique facility. It is not clear whether a polarized beam can be developed with sufficient intensity. Possibly a refraction separation of an intense unpolarized beam would work if not the use of reflection from a magnetic mirror. Nevertheless an intense beam is needed and can be developed with improvements of the monochromatic system. If we are to use the large detector, the expense of building a large sheilding can not be avoided. The very large active volume of the big detector will result in a tremendous background. If there is no improvement in incident intensity use of the large detector by itself will not be very useful.